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CHAMBER FOR CONTAMINATION SENSITIVE TESTS

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PREPARATION, VERIFICATION, AND OPERATIONAL CONTROL OF A LARGE SPACE-ENVIRONMENT-SIMULATION CHAMBER FOR CONTAMINATION SENSITIVE TESTS

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ABSTRACT

Thermal-vacuum testing of spacecraft carrying sensitive optical instruments poses special problems to the test facility with regard to contamination control during both preparation and test activities. A 2-year program to prepare chamber A at the NASA Manned Spacecraft Center for Apollo telescope mount thermal-vacuum tests is discussed. This program covers modification to existing chamber systems, the development of facility and chamber operating procedures, the selection of chamber cleaning and control methods, and the development and evaluation of diagnostic instrumentation. Specific details are included to illustrate the extensive efforts made to ensure a minimum contamination environment for the Apollo telescope mount test series. Recent thermal-vacuum tests of this chamber at progressive stages of cleanliness have demonstrated the success achieved in reducing the particulate and molecular contamination levels of the basic chamber interior. Data are presented that compare these "base line" contamination results.

INTRODUCTION

Contaminants may be introduced into a space-environment-simulation chamber during initial construction phases and during preparation and testing activities on spacecraft carrying sensitive optical instruments. Once introduced, these contaminants can cause severe detrimental effects on the instruments, such as a reduction in the reflectivity of mirrors and gratings of high-resolution vacuum ultraviolet (UV) spectrometers.

In relation to space-environment-simulation testing, contaminants are subdivided into two categories. The first category is particulate contamination that consists generally of dust particles, lint, rust, chafed particles from thermal and electrical insulator materials, and human epidermal cells. This form of contamination can present serious problems if it is not discovered and removed because particulates can degrade delicate electronic contacts, miniature mechanical systems, and block slits of spectrometers. Particulate contamination normally is experienced only during times when the chamber pressure is high enough to allow airborne transport; that is, during spacecraft preparation and checkout activities, during the initial chamber-pumpdown phase, and during the final phases of repressurization. The presence of particulates under high-vacuum (test) conditions seldom is experienced because there is no external source of particles and no matrix for the mobility of particles other than spacecraft waste dumps and the gravitational field.

The other category is molecular contamination. This form of contamination presents a more serious problem during high-vacuum conditions than particulate contamination. Molecular contamination consists of chemical substances and compounds that migrate and deposit in the chamber test volume as individual molecules or as clusters of molecules (ref. 1). When these substances condense on optical elements, they usually form two main types of contaminating surface layers: smooth and continuous layers of light absorbing and nonabsorbing materials, and surface condensations in the form of droplets that cause light scattering. The degradation caused by these contaminating surface layers normally is not permanent. When the source of contamination is removed, complete recovery usually occurs if the vacuum system is reasonably clean. However, it has been shown that restoration does not occur if a contaminated optical surface is irradiated with UV or other molecule-decomposing particles, such as electrons or protons (ref. 2). On the contrary, irradiation of contaminated mirrors with UV energy enhances the reflectance decrease and makes it permanent because the residue formed during the irradiation does not evaporate.

Materials that produce contaminating surface layers in a space-simulation chamber are derived from a myriad of sources. The most common sources, however, are backstreaming from mechanical pumps and oil-diffusion pumps; evaporation and sublimation of substances from warm chamber walls; desorption and outgassing products from paints, epoxies, and insulating

materials; and thermal decomposition of high-molecular-weight materials resulting in the release of light, volatile fractions.

The purpose of this report is to describe the overall program developed for chamber A at the NASA Manned Spacecraft Center (MSC) to reduce the particulate and molecular contamination levels during preparation and checkout activities for the Apollo telescope mount (ATM) and to maintain these levels within the test-environment requirements. This program covers modification to existing chamber systems, development of facility and chamber-operating procedures, selection of chamber cleaning and control methods, and development and evaluation of diagnostic instrumentation. Test results are presented that list principle contaminants and levels of concentration for three previous "base line" contamination-measurement tests. Conclusions and recommendations also are presented that review the present program in relation to future thermal-vacuum tests at the MSC.

FACILITY DESCRIPTION

Chamber A is a stainless-steel vessel 19.8 m in diameter and 36.6 m high. A side-hinged door, which provides a 12.2-m-diameter clear opening for vehicle loading, is located in the cylindrical section. The chamber is equipped with a rotating platform (lunar plane) for supporting test vehicles weighing up to 68 100 kg. Personnel access is provided from four manlocks and one chamber door. Double manlocks are provided at the lunar-plane level and the midchamber level. The access door is located at the upper chamber level.

The chamber interior is lined with black, liquid-nitrogen-cooled heat-sink panels that operate at approximately 90° K. The chamber vacuum system consists of a mechanical roughing system, eighteen 81-cm-diameter oil-diffusion pumps, and gaseous-helium-cooled cryopumping surfaces that operate at a temperature of approximately 17° K. Chamber pressure normally is maintained below 1×10^{-6} torr by the use of the helium cryopanel surfaces that produce pumping speeds for gases condensable on their surfaces in excess of 10 000 000 l/sec.

The chamber also is equipped with a top solar simulator system that consists of external carbon-arc light sources and internal collimating optics. The target area for the top sun system is 4 m in diameter. Within this area, the radiant energy intensity can be controlled in the range of 622 to 1353 w/m².

A larger "side" solar simulator is not used in ATM testing, and the penetration area for the light sources has been

covered temporarily by a liquid-nitrogen-cooled shroud that shields the ATM from the warm chamber wall.

Test-Vehicle Description

The ATM is an optical experiment payload consisting of six experiments designed specifically to study solar phenomena.

Experiment SO52 - White-Light Coronagraph--This experiment uses an externally occulted coronagraph to monitor, in the 4000 to 6000 Å wavelength range, the brightness, form, and polarization of the solar corona from 1.5 to 6 solar radii. It is used to study the evolution of coronal activity and to correlate this activity with events on the surface of the sun.

Experiment SO54 - X-Ray Spectrographic Telescope--This experiment records spectra of the solar flare X-ray emission in the 2- to 10-Å wavelength range with a resolution of 0.5 Å. It is used to follow the evolution of the spatial image and the spectrum of a flare throughout its lifetime and to observe the evolution of nonflaring active regions on the surface of the sun.

Experiment SO55A - UV Scanning Polychromator/Spectroheliometer--This experiment photoelectrically records high-resolution solar images in six spectral lines simultaneously and operates in the 300- to 1350-Å wavelength range. It is used to observe the solar atmosphere near or above active regions on the solar disk.

Experiment SO56 - Dual X-ray Telescope--This experiment obtains high-resolution (5 arc sec) photographs of the coronal X-ray emission of the sun in the 3- to 60-Å wavelength range. It is used to monitor active solar regions during both active and quiescent periods and to record total solar X-ray flux measurements.

Experiment SO82A - Extreme UV Coronal Spectroheliograph--This experiment obtains high resolution (5 arc sec) spectroheliograms of the solar atmosphere in the 150- to 650-Å wavelength range. It is used to provide an increased understanding of solar physics with possible practical application in the research of nuclear production.

Experiment SO82B - Extreme UV Spectrograph--This experiment records spectra of the solar disk in the 900- to 3900-Å wavelength range with a 0.08 to 0.16 Å spectral resolution. It

is used to record spectra from discrete areas of activity in the chromosphere of the sun.

The ATM provides a mounting structure, electrical power, fine pointing system, and environmental control for these experiments. It weighs 11 200 kg and is 4.39 m in length and 4.37 m in diameter. The ATM test configuration is shown in Figure 1.

Cleanliness Requirements

Tests on the ATM thermal systems unit and prototype vehicle were performed in chamber A during July and August of 1970 and October and November of 1971, respectively. Tests on the ATM flight vehicle presently are scheduled to be conducted during June and July of 1972. To protect the sensitive optical experiments on these vehicles from particulate and molecular contamination, the following rigid set of cleanliness specifications have been established by the NASA.

1. The chamber environment surrounding the ATM vehicle during preparation and checkout activities must approach the requirements of a conventional class 10 000 clean room (ref. 3). A class 10 000 clean-room environment is defined as an environment that has no more than 10 000 particles per 28.3 l of air larger than 0.5μ in size and not more than 65 particles per 28.3 l of air greater than 5μ in size.

2. Under thermal-vacuum test conditions, optical degradation for a first-surface aluminized mirror maintained at 20°C and irradiated with the Lyman Alpha hydrogen line (1216 \AA radiation) shall be less than 5 percent for the solar irradiation test phase (40-hr duration) and less than 10 percent for the entire test (70-hr duration).

To meet these requirements, extensive modifications had to be made to the chamber systems and operating procedures.

CHAMBER PREPARATION

Hardware and Procedural Modifications

Oil-Diffusion Pumps--It has been assumed previously that the most probable source for molecular contamination in vacuum chambers is oil-diffusion pumps (ref. 4). When a diffusion pump is fitted with an optically dense baffle, visible films of condensed oil above the baffle usually are prevented but contamination of the system by molecular film still can occur (ref. 5). The causes of such contamination may be summarized as

reevaporation of oil condensed on the baffle, direct penetration of the baffle after intermolecular collisions, and migration along warm boundary walls with subsequent reevaporation into the system.

To limit the quantity of molecular contamination introduced into the chamber by the oil-diffusion pumps, three corrective actions were used. The first action was to operate only four of the 18 pumps on the chamber and to use these four pumps for removing the noncondensable gas-leak loads. Calculations revealed that the combined pumping speed of these diffusion pumps would be satisfactory for removing the small "noncondensable" ATM gas load from the chamber. The second corrective action was to install liquid-nitrogen-cooled baffles between these four pumps and the angle valves that connect the pumps to the chambers. The baffles used are designed to provide optimum protection against the causes for diffusion-pump oil contamination. Each baffle is equipped with an anti-oil migration barrier and uses a double-bounce, optically tight chevron configuration. As a final corrective action, a procedural change was made to restrict operation of the oil-diffusion pumps to chamber pressures below 5×10^{-3} torr rather than the previous procedural upper limit of 5×10^{-2} torr to minimize direct penetration of the baffle by high-temperature oil molecules following intermolecular collisions.

After the liquid-nitrogen baffles were installed, tests were performed on one diffusion pump to measure the backstreaming rate under normal operating conditions. The average backstreaming rate was determined to be 5.17×10^{-9} g/cm²/day, or approximately 0.04 monolayers/day (ref. 6). As a result of these tests, it was decided that the diffusion pumps would not degrade the cleanliness levels inside the chamber because the backstreaming rates were low. Moreover, liquid-nitrogen heat-sink panels directly in front of the port openings, which were not considered in the backstreaming measurements, would be cooled to cryogenic temperatures before the diffusion pumps were opened to the chamber, providing additional assurance that oil would not reach the test volume.

Mechanical Pumping System--Corrective actions also were employed to limit the quantity of contamination introduced into the chamber by the mechanical (roughing) pumping system. These actions included installation of a liquid-nitrogen baffle in the roughing duct between the chamber isolation valve and the mechanical pumps and blowers, and a procedural change to close

the chamber isolation valve between the mechanical pumps and the chamber at a pressure of 1×10^{-3} torr, rather than at 5×10^{-4} torr as was done previously. To minimize backstreaming from the mechanical pumping system, cooldown of the baffle during pumpdown is initiated at a chamber pressure of 1 torr.

Air Locks and Chamber-Air-Recirculation System-- Facility modifications also were required to limit the quantity of particulate contamination introduced into the chamber. A cleanliness requirement established by the NASA was to approach a conventional class 10 000 clean-room environment inside the chamber during ATM preparation and checkout activities. To meet these limitations, the following corrective actions were used.

1. Double door air locks were installed at all laboratory entrances and maintained at a slightly positive pressure (0.4 torr) to limit the amount of particulate contamination introduced into the laboratory high-bay area.

2. Air showers were installed at all chamber personnel entrances to limit the quantity of contamination introduced into the chamber by test-team personnel.

3. All test-team personnel were required to dress in lint-free clothing before entering the chamber through the manlocks and upper level door.

4. The chamber-air-recirculation system was upgraded substantially to remove particulate contamination by providing a 99.97-percent effective filtration capability for particles 0.3μ in size or larger. This capability was established by adding prefilters that were 85 percent effective in removing particles 5μ in size or larger and high efficiency particulate air filters that trapped particles not removed by the prefilters. A mechanical blower was used to maintain the chamber pressure slightly above ambient pressure (0.4 torr) to limit the quantity of particulate contamination introduced into the chamber through the personnel entrances. The volumetric flow of air through the chamber when the recirculation system was in operation was

5.6×10^5 l/min. The recirculation system also was used to maintain the relative humidity inside the chamber below 50 percent when the vehicle loading door was closed. A schematic showing the chamber A air-recirculation system is included in Figure 2.

Chamber Cleaning and Preparation

Particulate contamination levels were controlled during ambient conditions by modifying the chamber-air-recirculation system to maintain a conventional class 10 000 clean-room environment inside the chamber. Molecular contamination, however, is more difficult to control and adheres to surfaces more tenaciously than particulate contamination. Two main types of molecular contaminants generally are observed in relation to the way these materials adhere to surfaces: those that exhibit a random molecular arrangement in the monolayer adjacent to the surface and those that show a definite preferred molecular orientation in the final monolayer (ref. 7).

The first class of materials, such as paraffin waxes and mineral oils, do not possess strong dipole moments and are retained on surfaces by weak Van der Waal forces and unsaturated metal forces. These materials are removed easily from substrates such as metals and optical elements by flushing the surfaces with reagents such as trichloroethylene and carbon tetrachloride. Molecular contaminants that exhibit a definite preferred orientation on surfaces such as diffusion- and mechanical-pump oils; greases, and residues of various cutting, lubricating, and cooling formulations possess strong dipole moments, and monolayers become tightly bonded to the substrates. If a sufficient amount of these materials is absorbed, the molecules in the final monolayer will align with one portion standing fairly erect. As the thickness of the film is increased beyond a monolayer, the orientation of the molecules becomes increasingly random. Molecules that exhibit preferred orientation in the final monolayer do so because such an arrangement is more stable than a randomly oriented system.

A program was implemented to select a detergent solution best suited to remove both kinds of molecular contaminants from the interior chamber surfaces. A total of four detergent solutions were tested. The detergent selected was found to be the most efficient in removing such tightly bonded substances as silicone oils, hydrocarbons, and phosphate ester fluids from surfaces, leaving behind a minimum of residue after the surfaces were rinsed with deionized water. These tests, the results of which are listed in Table I, consisted of contaminating quartz disks with $2 \times 10^{-5} \text{ g/cm}^2$ of sample and washing the disks with 50 ml of detergent solution. The disks were allowed to drain dry and were rinsed with 50 ml of boiling deionized water. The quantity of contamination remaining on the surfaces was determined by measuring the transmittance losses of the quartz disks in the near UV wavelength region.

The chamber-cleaning operation consisted of directing the detergent solution (one part detergent to 200 parts deionized water) onto the interior chamber surfaces using high-pressure hoses. The chamber was cleaned from the top down. A total of 18 900 l of detergent solution was used to clean the chamber. The chamber surfaces were then rinsed using 189 000 l of deionized water.

After the chamber was cleaned and outgassed to remove traces of moisture, particulate measurements were made and wipe samples were obtained from representative locations throughout the chamber and were analyzed to verify that only minute traces of contaminants and detergent residue remained. The results of the particulate measurements under various operating conditions, before and after chamber cleaning, are shown in Table II. Before cleaning the chamber, a conventional class 10 000 clean-room environment could not be maintained with 12 or more persons in the chamber with the recirculation blower on and the vehicle loading door closed (Tab. II). However, subsequent to chamber cleaning, the ambient environment was maintained well within the specifications for a conventional class 10 000 clean room. The results of the wipe-sample analysis are summarized in Table III. The molecular-contamination levels were reduced significantly as a result of chamber cleaning (Tab. III). These results revealed that the cleanliness level of the chamber interior now was satisfactory for proceeding with the contamination-verification test program.

To remove any contaminants introduced during preparation and checkout activities, it has become common practice to perform a pretest bakeout of the chamber. This is accomplished by increasing the lunar plane and liquid-nitrogen shrouds to a temperature of 40° C and operating the chamber at a pressure less than 5×10^{-3} torr for a minimum period of 72 hr. The temperature and pressure parameters were selected because they were within the operational limitations of the chamber. The increased temperature and reduced pressure increased the outgassing rates of materials in the chamber interior. The time duration was selected because it has been shown that the outgassing rates for many polymers and metal surfaces decrease exponentially with time (ref. 8), and that at the end of the period, the outgassing rates would be reduced to a level that would not be detrimental to the ATM optical experiments.

Material Selection and Control

Before chamber cleaning was accomplished, a material selection and control program was implemented to minimize recontamination of the interior chamber walls and cryopanel surfaces by introduction of polymeric materials that have high outgassing rates at elevated (125°C or greater) temperatures. The criterion used at the MSC for material selection is based on the quantity of volatile condensable material and weight loss of a material sample maintained under controlled conditions (ref. 9). A material sample is rated as acceptable if it has a maximum volatile condensable material of 0.1 percent and a total weight loss of 1.0 percent when tested under a pressure of 10^{-6} torr or less, a specimen temperature of $+125^{\circ} \pm 1^{\circ}\text{C}$, a condensable plates temperature of $+25^{\circ} \pm 1^{\circ}\text{C}$, and a vacuum exposure time of 24 hr.

As part of a continuing program to refine and extend the material-selection criteria, an additional technique for material evaluation is being considered by the MSC. This technique would require material specimens to be tested under conditions similar to those previously mentioned and irradiated with UV energy. Materials otherwise acceptable would be disapproved for vacuum-chamber application if UV irradiation caused unacceptable increases in weight loss and quantity of volatile condensable materials. Acceptance criteria for this technique has not been established at the present time.

A material-usage-control board has been established by the MSC that has jurisdiction over all materials to be installed in the test chambers. Materials that have been tested but failed to meet the requirements just mentioned may be used if the performing organization can provide rationale for its use that is approved by this board. Usually, materials are approved that fail to meet the selection requirements if the material is the best available for the particular application; the quantity and surface area of the material is small, and not in the immediate vicinity of an optical element; and the material may be vacuum cured before use.

As a final safeguard against contamination introduction, all polymeric materials used in chamber A for the first time must be preprocessed by exposure to a room-temperature environment and maintained at a pressure less than 1×10^{-5} torr for at least 4 hr. All materials (including polymeric materials and metal assemblies) must be cleaned thoroughly with either

Freon TF or ethyl alcohol, inspected using the black-light technique, and wrapped in clean-room bags for handling before installation in the test chamber.

DIAGNOSTIC INSTRUMENTATION

Three basic objectives for contamination measurement in space-simulation chambers are to determine the effects of contamination in relation to sensitive experiments on the test vehicle, to identify the contaminants and determine their concentrations (qualitative and quantitative analysis), and to determine the sources of contamination. Several groups of instruments are used by the MSC and the NASA George C. Marshall Space Flight Center (MSFC) in the ATM program for accomplishing these objectives.

Vacuum Ultraviolet Reflectometers

An in situ reflectometer, or real-time contamination monitor (RTCM), was developed to monitor the degree of optical degradation resulting from the formation of surface films during thermal-vacuum tests. This instrument consists of a radiation source, detectors, and a first-surface (Al + MgF₂) mirror

mounted on an optical bench. The system essentially is monochromatic and monitors reflectance changes on the mirror using the 1216-Å line (Lyman Alpha) emitted by the radiation source. A detailed description of this instrument is found in reference 2. Locations and operating environments for this instrument are shown in Figure 3 and Table IV. One of the reflectometers is shown in Figure 4.

Residual Gas Analyzers and Ionization Gages

Three systems are used for qualitative analysis of gaseous contaminants in the chamber-vacuum environment: monopole residual-gas analyzers, quadrupole residual gas analyzers and direction gas flow measurement (DGFM) systems. Locations and operating environments for these instruments are shown in Figure 3 and Table IV.

The monopole and quadrupole residual gas analyzers are standard instruments modified for use in large space-simulation chambers. Modifications to the quadrupole residual-gas analyzers included lengthening the electric cables between the analyzer head and control console to approximately 17 m, installing a special low-noise preamplifier at the analyzer head, and

changing mass coils for the radio frequency rf/dc generator inside the control unit. Modification to the monopole residual-gas analyzer (RGA) included lengthening the electric cables between the analyzer head and control unit and modifying the electronics within the radio frequency tank circuit. One of the quadrupole residual-gas analyzers is shown in Figure 4.

The DGFM System--The DGFM system is used to identify chamber leak sources and residual gases in the chamber environment. It also is used to determine locations of contaminant sources within the chamber. This system consists of two units. The first unit includes two pairs of ionization gages (one pair of redundancy) and a quadrupole RGA mounted on a rotatable platform within the chamber. The second system is identical to the first except that two millitorr gages are used in place of the quadrupole RGA and are used to locate chamber leak sources at higher chamber pressures (10^{-2} torr to 10^{-4} torr). The pressure gages and quadrupole RGA are equipped with tubulations to provide more accurate angular definition of a directional gas flow source. A detailed description of the DGFM is found in reference 10.

Quartz Crystal Microbalances

Two kinds of quartz-crystal microbalances are used to measure the quantity of molecular contamination that condenses on the reflectometer mirrors to produce reflectance changes. The first unit is a deposit thickness monitor (DTM) and the second unit is a quartz-crystal microbalance contamination monitor (QCM/CM). Locations and operating environments for these instruments are listed in Figure 3 and Table IV.

Both quartz-crystal microbalances basically consist of a sensing crystal, a reference crystal, and electronics for driving the crystals and producing an audio-beat frequency by mixing the fundamental crystal frequencies. The DTM has a sensing crystal located on the oscillator unit inside the chamber and a reference crystal located in the control unit outside the chamber. The QCM/CM has both crystals located in the oscillator unit inside the chamber.

The sensing crystal for both quartz-crystal microbalances is tuned initially below the reference crystal. As contamination condenses on the sensing crystal, it lowers its resonant frequency and, in turn, causes the frequency difference, or beat frequency, to increase. Changes in beat frequency are recorded because they are directly proportional to changes in mass on the sensing-crystal surface.

Analytical-Test Specimens

Three kinds of analytical specimens are used to obtain quantitative and qualitative information concerning condensable chamber contaminants: contamination-collection units, stainless-steel disks, and aluminum disks. Locations and operating environments for these specimens are listed in Figure 3 and Table IV.

Contamination-Collection Units--The contamination-collection units (CCU) are passive-sampling devices and are used to collect condensable contaminants from the chamber environment for post-test analysis. These specimens consist of a 30 by 30 by 0.5-cm Pyrex glass plate mounted in an aluminum frame. Fifteen contamination-collection units were used during recent tests. Three of the contamination-collection units are mounted in a temperature controlled ($5^{\circ} \pm 1^{\circ}$ C) test bed and are exposed individually during chamber pumpdown, test, and repressurization. The remaining contamination-collection units are not temperature controlled and are allowed to cool until they reach thermal equilibrium with the chamber-heat-sink panels. After the test is completed, the contamination-collection units are removed from the chamber, and contaminants are collected, identified, and concentrated for infrared- and gas-chromatographic analysis. A detailed description of contamination-collection unit analysis techniques is found in reference 11.

Stainless-Steel and Aluminum Disks--These test specimens also are passive-sampling devices and consist of two types that are exposed to the chamber environment at two temperatures. One group of specimens is mounted on two temperature controlled ($5^{\circ} \pm 1^{\circ}$ C) test beds. One test bed exposed the specimens to the chamber environment for the duration of the test. The other test bed is equipped with a four-section shutter arrangement and is controlled remotely to expose four different sets of specimens sequentially to the chamber environment for specific test phases (pumpdown, high vacuum, solar, and repressurization). The other group of specimens is placed in special holders or mounted individually at various locations within the chamber such as roughing lines and pump ports. These specimens are not temperature controlled. The deposits collected on both groups of disks are analyzed with infrared spectrophotometers and residual-gas analyzers after the test is completed.

Optical Test Specimens

These test specimens also are passive-sampling devices and consist of four types: quartz disks, Al + MgF₂ mirrors, platinum mirrors, and gold mirrors. These specimens are mounted on the temperature controlled test beds and also in the special holders that are not temperature controlled. The quartz disks and Al + MgF₂ mirrors also are mounted on selected contamination-collection units within the chamber. These specimens are removed from the chamber after the test is completed. Transmittance and reflectance measurements are made on the quartz disks and mirrors, respectively, to determine the degree of optical degradation subsequent to exposure to the test environment. An optical test bed is shown in Figure 5.

Test Results

Three contamination-verification tests, identified as tests V-1, V-2, and V-3, were conducted in chamber A at progressive stages of cleanliness to determine molecular-contamination levels present during test conditions. These conditions were defined as a chamber pressure of 1×10^{-5} torr or less and a liquid-nitrogen heat-sink panel temperature of 100° K or less. During these tests, the top solar simulator system was operated both intermittently and continuously at an intensity level of one-solar constant. The duration of the test phase, including solar-simulator operation, is listed for each test in Table V.

Test V-1 was conducted in December 1969 and was a base line test of the empty chamber to evaluate the contamination levels after system and procedural modifications previously discussed were incorporated. Test V-2 was performed during May 1970 to verify that the corrective actions required, subsequent to the V-1 test, had reduced the contamination levels for the chamber. Test V-3 was performed during July 1971, to verify that the chamber was still at the level of cleanliness obtained during the V-2 test and that the addition of such ATM support equipment as the infrared simulator and UV calibration sources did not degrade the clean environment.

Test V-1 - Contamination Verification

The molecular-contamination levels measured during this test were higher than for subsequent tests, although the test environment did meet the cleanliness requirements established

by the NASA. The test results are summarized in Table V. The average reflectance loss for the MSFC aluminized mirrors at a wavelength of 1216 Å was 7 percent. The maximum reflectance loss for these mirrors occurred at a wavelength of 2000 Å and was 15 percent. There was good correlation between this value and the reflectance losses recorded at this wavelength for the MSC aluminized mirrors that was 16 percent. A comparison between these data and the real-time reflectometer measurements could not be made because the reflectometers became inoperative early in the test. In addition to loss of the reflectometer data, no data were obtained from the residual-gas analyzers because these instruments also became inoperative early in the test.

The correlation obtained between the QCM real-time measurements and the CCU post-test analysis results were good also. The quartz-crystal microbalances were maintained at approximately 0° C during the test, whereas, the temperature of the contamination-collection units was uncontrolled. The quartz-crystal microbalances recorded an overall contamination level of 8×10^{-7} g/cm². The CCU analysis results indicated an average contamination level of 6×10^{-7} g/cm².

The dominant contaminants, as determined from analysis of the contamination-collection units, consisted of paraffinic hydrocarbons and alkyl phthalate esters. Silicones also were present on the contamination-collection units in trace amounts. The paraffinic hydrocarbons have saturated or slightly unsaturated long-carbon atom chains and are similar in many respects to mineral oil. The sources for these contaminants include human-skin oils, lubricant used on the diffusion-pump angle-valve shafts, various cutting oils and cooling formulations, and outgassing products from epoxy paints. The alkyl phthalate esters are typical components of tall oil alkyd resin-based paints. Heavy esters, alkyd resins, drying oils, and alcoholic ethers (cellosolves) are used as matrices for these paints and are the major outgassed materials (ref. 12). The most likely source for this contaminant is the black epoxy paint used on the chamber-heat-sink panels and lunar plane. The silicones are of the polydialkylsiloxane type and are similar to polymers of dimethylsiloxane. These silicones are different in chemical structure from silicone diffusion-pump oil that is a methyl-phenyltrisiloxane compound. Sources for the dimethylsiloxane include silicone oils, greases used on "O"-ring seals, rubbers, and sealants.

Analysis results of the analytical specimens indicated that mechanical-(roughing) pump fluids also were present in the test volume. Diffusion-pump oil was not found on the

contamination-collection units or analytical specimens in the test volume. This fluid was, however, found on the contamination-collection units placed behind the chamber heat-sink panels directly in front of the diffusion-pump ports. Trace amounts of this fluid were present in addition to the hydrocarbon lubricant used on the angle-valve shafts. The average contamination level for the contamination-collection units at this location was $10 \times 10^{-7} \text{ g/cm}^2$.

Corrective Actions

To reduce the contamination levels measured during the V-1 test and to maintain these levels below the acceptable limits for the ATM test program, several corrective actions were employed. First, unused mechanical fluid lines and electrical conduits inside the chamber were either removed or capped to limit contamination resulting from evaporation of oil films inside the fluid lines and outgassing of electrical wiring insulation inside the conduit. Second, materials inside the chamber such as electrical cables, wiring harnesses, cable ties, and tape were reviewed for outgassing properties. Materials that were unacceptable were either removed or replaced. Third, the common practice of lubricating O-ring seals with silicone oil or grease was discontinued. A new procedure was established that required these seals to be installed dry. Subsequent tests revealed this procedure did not adversely affect seal reliability. Finally, traces of grease and oil deposited on interior chamber surfaces during leak-checking operations were discovered and removed. A second contamination-verification test was performed after these corrective actions were implemented.

Test V-2 - Contamination Verification

The molecular-contamination levels measured during this test were lower than for the previous test and were within the cleanliness requirements established by the NASA for the ATM test program. The test results are summarized in Table V. The maximum reflectance loss was recorded for the MSC aluminized mirrors and was 14 percent. The reflectance loss recorded for the real-time reflectometer during the high-vacuum phase was 10 percent. However, these measurements are not representative of the cleanliness level of the chamber because reflectance losses for the MSC aluminized mirrors were not recorded at a wavelength of 1216 Å, and the real-time reflectometer was not maintained at a temperature near 20° C

as required by the acceptance criteria. Reflectance losses for the MSC mirrors were recorded at a wavelength of 2000 Å. The reflectometer showed a loss in reflectance when its mirror was cold and an increase in reflectance when it was warmed by radiation from the top solar simulator system.

The least reflectance losses were recorded for the MSFC aluminized mirrors on the test beds and are representative of the cleanliness levels of the chamber because reflectance measurements were made at wavelengths that included 1216 Å, and the mirrors were maintained at a temperature near 20° C during the test. The average reflectance loss recorded for the test bed mirrors exposed for the duration of the test was 4.9 percent. The average reflectance loss recorded for the test bed mirrors exposed during the solar phase and high-vacuum phase was 2.3 and 4.1 percent, respectively.

During this test, real-time gas-analysis data were recorded with the MSFC monopole RGA. The other residual-gas analyzers either became inoperative or malfunctioned, and presentable data were not obtained from them. Mass scans recorded with the monopole RGA primarily indicated an air leak, and occasionally higher molecular-weight peaks appeared that indicated the presence of alcohol. This compound probably was released when the helium cryopanel inadvertently warmed during the test. These peaks persisted for only a few hours. The presence of alcohol was attributed to cleaning agents used in the chamber before the test.

The correlation obtained between the QCM real-time measurements and the CCU post-test analysis results was again in good agreement. The quartz-crystal microbalances recorded an overall contamination level of 4×10^{-7} g/cm². The CCU analysis results indicated an average contamination level of 3×10^{-7} g/cm² for passive and temperature controlled contamination-collection units.

The dominant contaminant as determined from analysis of the contamination-collection units was a paraffinic hydrocarbon. Trace amounts of organic esters also were present; however, these substances were unidentifiable because of low concentrations. Evidence of mechanical-pump oils, silicone oils, and greases also were found on the contamination-collection units in extremely small concentrations. The temperature controlled contamination-collection units indicated the greatest quantity of contamination was deposited during chamber repressurization. Approximately 4.5×10^{-7} g/cm² was collected during this phase, whereas, 1.83×10^{-7} and 0.14×10^{-7} g/cm² were deposited during pumpdown and test phases, respectively. Diffusion-pump

oil did not appear on any of the contamination-collection units in the chamber. The contaminants on the contamination-collection units facing the diffusion-pump ports were predominately hydrocarbons. The average contamination level at this location was approximately 4.3×10^{-7} g/cm².

Trace amounts of contaminants also were found on the analytical specimens located in the test volume. Only one specimen showed a significant amount of contamination. This particular sample was located between the chamber wall and heat-sink panels and collected approximately 3.4 mg of hydrocarbons.

Test V-3 - Contamination Verification

The molecular-contamination levels measured during this test were lower than for the two previous tests. These results indicated the cleanliness levels for the chamber were not degraded by the addition of the ATM support equipment. The test results are summarized in Table V. The real-time reflectometer showed a slight reflectance loss during the solar phase but gradually recovered during the remainder of the high-vacuum phase. This recovery is attributed to sublimation or evaporation of surface contaminants that condensed on the reflectometer mirror during the solar phase.

Optical data for the MSFC test bed specimens yielded reflectance values that were within the limits of the test criteria for all samples, except those under shutter no. 2 (high vacuum), which yielded marginal data. This was consistent with the results obtained in the V-2 test in that samples under shutter no. 2 showed the largest loss in reflectance. The reflectance losses for these samples were measured at 1600 Å because this is the strongest line in the hydrogen spectrum. Reflectance losses for the MSC samples were measured at 2000 Å and compared favorably with the losses for the samples placed under shutter no. 2 (12 percent and 11 percent, respectively). Again, these values do not relate directly to cleanliness acceptance criteria, inasmuch as the reflectance measurements were not recorded at a wavelength of 1216 Å.

The residual gas analyzers operated satisfactorily during this test, and data obtained with these instruments indicated that most molecular activity occurred during the solar phase when shutter no. 3 on the test bed was open. The activity began approximately 1 hr after initiation of the continuous solar burn and terminated 30 min after this phase was completed. These data are consistent with reflectance losses recorded with the real-time reflectometer that indicated maximum degradation occurred during the solar phase of the test cycle. These losses are attributed to the formation of surface films that occurred

when molecular contaminants were released from chamber surfaces warmed by the solar energy.

The correlation between the QCM real-time measurements and the CCU post-test analysis results was again in good agreement. The data listed in Table V for the temperature controlled contamination collection units indicated that maximum contamination occurred during pumpdown. Approximately

$0.32 \times 10^{-7} \text{ g/cm}^2$ was collected during this phase, whereas, an insignificant amount was deposited on the contamination-collection units during the high-vacuum phase and repressurization. The quartz-crystal microbalances also measured contamination during pumpdown. Contaminants that deposited on these instruments during this time began to slowly evolve once test conditions were achieved. The quartz-crystal microbalances continued to recover throughout the high-vacuum phase.

The CCU analysis results revealed that the contaminants in the test volume were predominantly hydrocarbons. Trace amounts of esters and silicones (including diffusion-pump oil) also were present on the CCU. The contamination-collection units facing the diffusion-pump ports collected the greatest quantity of contamination. Traces of diffusion-pump oil also were found on these contamination collection units. The average quantity of contamination for the CCU at this location was

$2.75 \times 10^{-7} \text{ g/cm}^2$, whereas, the test volume average was $0.84 \times 10^{-7} \text{ g/cm}^2$.

The CCU near the real-time reflectometers indicated a contamination level that was much greater than the average throughout the chamber. A quantity of $1.3 \times 10^{-7} \text{ g/cm}^2$ was measured at this location. The quartz disk on this unit indicated severe optical degradation (-49 percent). The predominant contaminant was found to be tape residue. A severe reflectance loss was observed for the MSC real-time reflectometer during chamber repressurization. During this time, the mirror reflectance fell from approximately 71 percent to 35 percent. These losses were attributed to condensation of tape residue that was released under high temperatures when electrical power cables for the ATM infrared simulator began to inadvertently arc or short circuit during repressurization.

Summary and Conclusions

The results of the last two contamination-verification tests revealed that a large thermal-vacuum test chamber can be

operated to produce a minimum contamination environment suitable for testing advanced spacecraft carrying sensitive optical experiments. With the measurement techniques used, it has been possible to identify partially and to measure accurately contaminants present under high-vacuum conditions during any specific test. Several general observations may be made as a result of these tests.

1. Contamination traceable to the four oil-diffusion pumps was not detected in the shrouded chamber volume during the first two tests. Only slight traces were detected during the last test.

2. Measurements obtained from the optical samples and real-time reflectometer indicate that chamber A is acceptable for Apollo telescope mount use according to the established test criteria.

3. Residual-gas-analyzer data detected gaseous molecular contaminants within the shrouded chamber volume at various phases during the test cycle. Most molecular activity occurred during the continuous solar burn. The optical samples exposed during this time experienced minor reflectance losses. The contaminants present are either transparent to the wavelengths being scanned or are not condensing on the Al + MgF₂ surfaces.

Future spacecraft may require more stringent contamination requirements and functional testing much longer than experienced at the present time. This will impose a severe constraint on environmental test engineers in selection of chamber materials and development of new diagnostic instrumentation. Future tests will require assurance that the test facility is clean and that the test environment presents a minimum contamination hazard.

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TABLE I. - EVALUATION OF DETERGENT SOLUTIONS FOR CHAMBER A CLEANING APPLICATION^a

Detergent	Test fluid				
	Houghto-Safe 1055 ^b	Houghto-Safe 1120 ^b	SunVis 706 ^c	DC 705 ^d	DC 11 ^e
No. 1 (boiling) percent remaining weight, g/cm ²	0.89 percent 1.7 × 10 ⁻⁷	0.79 percent 1.6 × 10 ⁻⁷	5.0 percent 0.1 × 10 ⁻⁶	0.07 percent 0.1 × 10 ⁻⁷	116 percent 2.3 × 10 ⁻⁵
No. 2 (boiling) percent remaining weight, g/cm ²	0.66 percent 1.3 × 10 ⁻⁷	0.86 percent 1.7 × 10 ⁻⁷	5.8 percent 1.2 × 10 ⁻⁶	0.47 percent 1.0 × 10 ⁻⁷	67.7 percent 1.4 × 10 ⁻⁵
No. 3 (boiling) percent remaining weight, g/cm ²	0.53 percent 1.0 × 10 ⁻⁷	0.69 percent 1.4 × 10 ⁻⁷	4.3 percent 9.0 × 10 ⁻⁷	0.22 percent 0.4 × 10 ⁻⁷	6.4 percent 1.3 × 10 ⁻⁶
No. 4 (room temp.) percent remaining weight, g/cm ²	0.66 percent 1.3 × 10 ⁻⁷	0.40 percent 0.8 × 10 ⁻⁷	4.3 percent 9.0 × 10 ⁻⁷	0.17 percent 0.3 × 10 ⁻⁷	6.4 percent 1.3 × 10 ⁻⁶

^a Quartz disks contaminated with 2 × 10⁻⁵ g/cm² of sample.

^b Houghto-Safe 1055 and 1120 are phosphate ester fluids used as mechanical-pump oil.

^c SunVis 706 is a hydrocarbon used as hydraulic fluid for the diffusion-pump angle valves.

^d DC 705 is a silicone diffusion-pump oil.

^e DC 11 is a silicone grease used as an O-ring sealant.

^f Mass increase is a result of soap residue remaining on contaminated disk.

TABLE II. - CHAMBER A PARTICULATE CONTAMINATION
LEVELS FOR AMBIENT ENVIRONMENTS

Chamber status	Before cleaning		After cleaning	
	Particles/28.3 l			
	>0.5 μ	>5.0 μ	>0.5 μ	>5.0 μ
Access doors closed - recirculation blower on - one man in the chamber	9 000	80	800	10
Access doors closed - recirculation blower on - 12 men in the chamber	17 960	231	1 400	10

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TABLE III. - WIPE-SAMPLE ANALYSIS RESULTS FOR REPRESENTATIVE CHAMBER A

INTERIOR LOCATIONS

Wipe-sample location	Contamination (10^{-7} g/cm ²)							
	Before cleaning:				After cleaning			
	H/C ^a	Ester ^b	Silicone ^c	D/P fluid ^d	H/C ^a	Ester ^b	Silicone ^c	D/P fluid ^d
Backside of liquid nitrogen panel, facing diffusion-pump port	20.0	6.0		18.0	1.4			3.0
Front side of liquid nitrogen panel midchamber level, southeast side	3.6	1.3						
Front side of liquid nitrogen panel midchamber level, southwest side	5.5	2.4			5.6	0.82		
Roughing duct entrance	34.0	23.0	1.9	2.5	8.9	17.0	trace	
Chamber wall, below lunar plane	11.0	5.4	9.3		1.0			

^aH/C - paraffinic hydrocarbon.

^bEster - alkyl phthalate ester.

^cSilicone - polydialkylsiloxanes.

^dD/P fluid - diffusion-pump fluid.

TABLE IV. - CHAMBER A CONTAMINATION-MEASUREMENT SYSTEMS

System	Instrument	Quantity		Operating conditions		Purpose
		MSC	MSFC	Pressure	Temperature	
Reflectometer	RTCM	1	3	$<10^{-3}$ torr (P, T) ^{a, c}	20° C	Optical effects measurement
				<600 torr (R) ^b	20° C (1) Uncontrolled (2)	
Total and partial pressure analyzers	DGFM	2		$<10^{-3}$ torr	N/A	Directional gas flow analysis
	Monopole RCA		1	$<10^{-4}$ torr	N/A	Qualitative measurement
	Quadrupole RCA	1	2	$<10^{-4}$ torr	N/A	
	Ionization gages		3	$<10^{-4}$ torr	N/A	Total pressure measurement
Quartz-crystal microbalance	DTM		2	≤ 760 torr	5° C	Quantitative measurement
	QCM/CM	2	-	≤ 760 torr ≤ 760 torr	20° C Uncontrolled	
Analytical and optical test specimens	Passive contamination-collection units and analytical specimens	12	24	≤ 760 torr	Uncontrolled	Quantitative, qualitative, and effects measurement (post-test)
	Passive optical samples	8	24	≤ 760 torr	Uncontrolled	
	Temperature controlled contamination-collection units	1		$>10^{-5}$ torr (P) ^a	5° C	
		1		$<10^{-5}$ torr (T) ^c	5° C	
		1		$>10^{-5}$ torr (R)	5° C	
	Temperature controlled contamination-collection units optical samples	6		$>10^{-5}$ torr (R)	5° C	
	Open test bed optical samples and analytical specimens	8	24	≤ 760 torr	5° C	
	Shuttered test bed optical samples and analytical specimens	2	8	$>10^{-5}$ torr (P) ^a	5° C	
		2	8	$<10^{-5}$ torr (T) ^c	5° C	
		2	8	$<10^{-5}$ torr (S) ^d	5° C	
		2	8	$>10^{-5}$ torr (R) ^b	5° C	

^a(P) = Pumpdown.^b(R) = Repressurization.^c(T) = Test phase.^d(S) = Solar phase.

TABLE V. - CHAMBER A CONTAMINATION VERIFICATION TEST SUMMARY

Instrument	Chamber A ATM coplanation-measurement tests representative data		
	H/C> Ester>>Silicone ^a	H/C> Ester>>Silicone ^a	H/C> Ester>>Silicone ^a
	ATM V-3 (July 1971)	ATM V-2 (May 1970)	ATM V-1 (Dec. 1969)
I. RTCM - $\Delta R/R$ at 1216 Å Solar phase High-vacuum phase	-1.41 percent +0.70 percent	+4.1 percent -10.0 percent	(e)
II. MSC aluminum mirrors - $\Delta R/R$ at 2000 Å ^b Test volume average Passive CCU average	-12.2 percent -11.0 percent	-14.0 percent (f)	-16.0 percent (f)
III. MSFC aluminum mirrors - $\Delta R/R$, average ^b Shutter test bed Shutter no. 1 (roughing) Shutter no. 2 (high vacuum) Shutter no. 3 (solar) Shutter no. 4 (repressurization) Test bed at 1.5-m elevation Test bed at 5.8-m elevation	1600 Å -2.5 percent -11.0 percent -2.0 percent -1.0 percent -0.9 percent (f)	1150 Å 1216 Å -1.9 percent -2.4 percent -11.5 percent -4.1 percent -7.9 percent -2.3 percent -7.5 percent -3.45 percent -7.8 percent -4.25 percent -11.2 percent -5.55 percent	(f) 1216 Å 1600 Å 2000 Å -7 percent -5 percent -15 percent (f)
IV. QCM/CM - 10^{-7} g/cm ² Solar phase High-vacuum phase	-0.75 -0.25	+1.1 +4.0	+3.1 +8.0
V. MSC CCU analysis - 10^{-7} g/cm ² Test volume average Diffusion pump average CCU 1-3 (roughing) CCU 1-4 (high vacuum) CCU 1-5 (repressurization)	0.84 2.75 0.32 0.00 0.04	3.0 4.3 1.83 0.14 4.47	6.0 10.0 (f) (f) (f)
VI. MSC quartz disks - $\Delta T/T$ at 2000 Å ^c Test volume average Passive CCU average MSFC test bed average	-6.0 percent -5.7 percent -12.8 percent	-2.8 percent -2.7 percent -6.1 percent	-4.5 percent -4.5 percent -3.9 percent
VII. Test time, hr ^d	82 (40)	120 (31)	61 (31)

^a Dominant contaminants are listed for each test in decreasing order of concentration; H/C represents paraffinic hydrocarbons; ester-alkyl phthalate esters; and silicone-polydialkylsiloxanes.

^b $\Delta R/R$ is percent change in optical (specular) reflectance.

^c $\Delta T/T$ is percent change in optical transmittance.

^d First value is for time at high vacuum; second value is for time of solar operation.

^e Instrument inoperative during test.

^f Instrument/samples not used for test.

Fig. 1—Chamber A ATM
test configuration

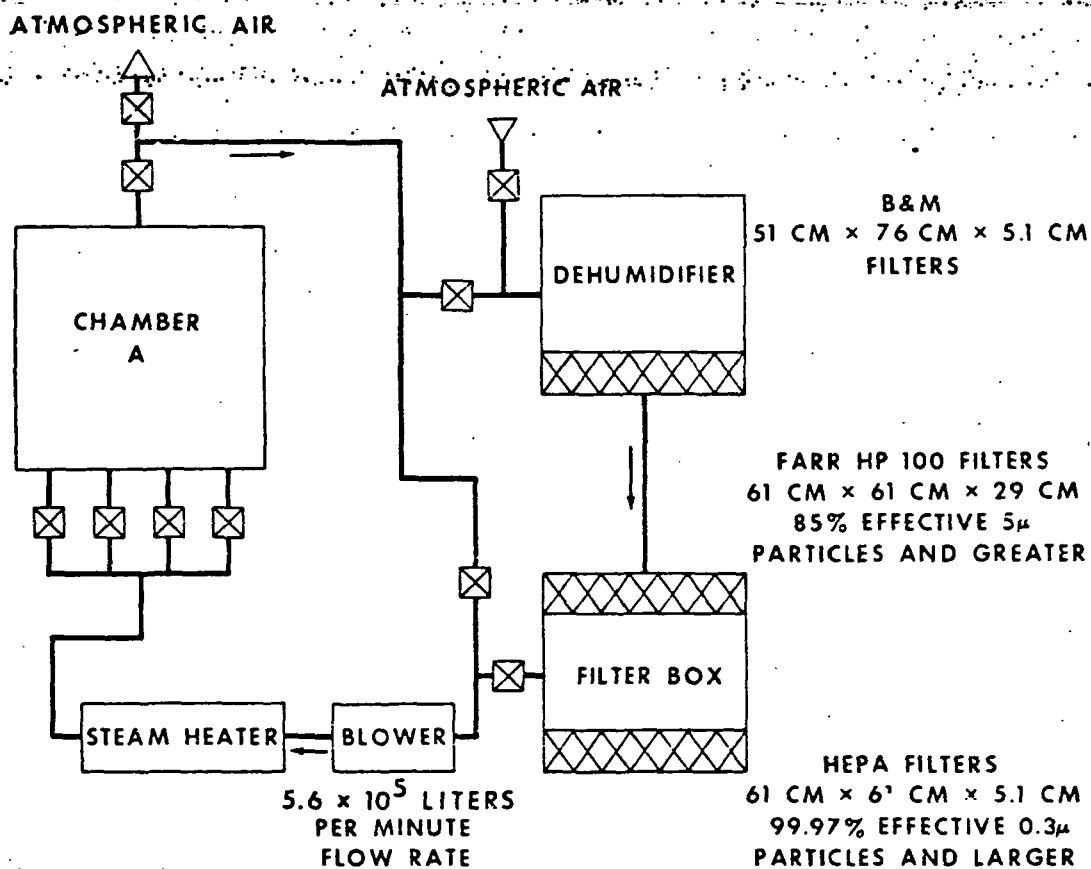


Fig. 2—Chamber A air recirculation system